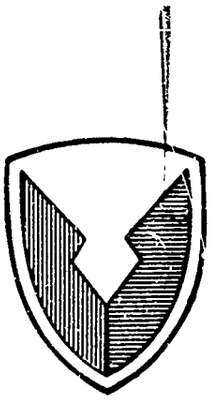

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CCL REPORT NO. 228

FINAL REPORT

POLYAMIDE-DIPHENOLIC ACID WASH PRIMER

BY

STANLEY F. KOUTEK

APRIL 1967

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U. S. ARMY COATING & CHEMICAL LABORATORY
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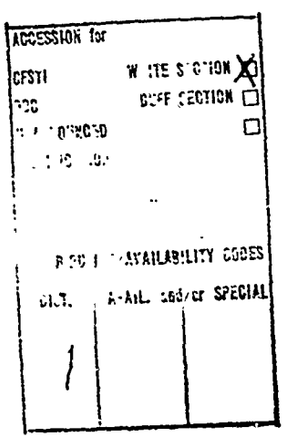

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U. S. ARMY COATING AND CHEMICAL LABORATORY
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ABSTRACT

A solid polyamide resin containing diphenolic acid was formulated into one and two package wash primers and evaluated against the control pretreatment primer, MIL-C-15328, for salt spray and water immersion resistance on steel, aluminum and magnesium substrates. Differences in performance between MIL-C-15328 and the two package diphenolic acid wash primer were negligible; however, the one package wash primers were inferior to the two package ones, particularly in water immersion.

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I. INTRODUCTION

Proper substrate preparation is necessary for the successful painting of metal surfaces. In addition to cleaning the metal, a surface treatment, such as a phosphate pretreatment on ferrous metals and chemical or electrolytic treatments on non-ferrous materials, is often employed for maximum corrosion protection. Frequently, a two component pretreatment wash primer, covered by specification MIL-C-15328 is used on these substrates, particularly in applications where special processing equipment required for applying the electrolytic and chemical conversion coatings is not available and/or practical. Although MIL-C-15328 enhances substrate protection and adhesion properties, field experience and laboratory studies indicate the need for improvement with respect to uniformity of performance and improved corrosion resistance on both ferrous and non-ferrous metals. Also desirable would be a one component wash primer. This would result in handling and storage savings as well as minimizing the chances of error in application for those not familiar with the two component system.

II. DETAILS OF TEST

The standard two component wash primer conforming to MIL-C-15328 (Table II) contains polyvinyl butyral as the vehicle. A two component wash primer using a polyamide-diphenolic acid resin as the binder is shown in Table III. According to the manufacturer's literature, this resin possesses outstanding adhesion to a variety of substrates, good resistance properties, rapid solvent release and is readily soluble in the alcohols normally employed in wash primers. Formulas C and D (Table IV and V) are one package materials using the polyamide-diphenolic acid resin at ratios of 4 parts vehicle solids to 1 part acid by weight and 6 parts vehicle solids to 1 part acid by weight, respectively.

Salt spray and aerated water immersion tests were selected as a means of evaluating the primers. The wash primers were applied by spray to a dry film thickness between 0.00025 and 0.0003 inch on 4 by 12 inch panels of ground steel conforming to Federal Test Method Standard No. 141, Method 2011, 24S Aluminum and AZ-31 Magnesium. The panels were vapor degreased and solvent cleaned in accordance with Method 2011 of of Federal Test Method Standard No. 141. MIL-C-15328 was applied to magnesium at 1/2 acid strength which is the standard practice to prevent hydrogen evolution. The experimental primers were tested using the formula acid concentration with no gassing observed on magnesium. After approximately 30 minutes air drying, the wash primers were topcoated with 0.001 inch dry film thickness of olive drab semi-gloss rust inhibiting enamel conforming to specification TT-E-485, Type II, and allowed to air dry 96 hours before testing. Panels for salt spray exposure were scored and exposed for a maximum of 500 hours to a 20 percent sodium chloride fog as in Method 6061 of Federal Test Method Standard No. 141, and rated in accordance with Table I. Unscored panels were immersed in aerated

water at 95° ± 2°F. and examined every 24 hours for a period of 120 hours. For package stability studies, the wash primers were stored at ambient temperatures in pint cans and checked periodically up to 4 months; in addition, the one package primers were stored in glass jars and viscosity determinations and flow outs on glass were made to examine for reactivity and seeding.

III. DISCUSSION

The two component polyamide-diphenolic acid resin primer (Formula B) contains a higher non-volatile content than MIL-C-15328 (Formula A) at a lower viscosity giving the advantage of a larger square foot coverage per gallon at comparable film thicknesses. Settling after 2 months storage was the same for both primers. One package wash primer (Formula C) exhibited seeding after 2 weeks and the viscosity increased steadily until a gummy state was attained in 2 months. In an attempt to improve stability, Formula D was prepared with a reduced phosphoric acid content. Viscosity determinations indicated no significant change after 4 months storage and no evidence of seeding was observed. Table VI lists the physical properties of the wash primers.

Salt spray exposure (Table VII) shows the two component polyamide-phenolic (primer B) to be comparable to MIL-C-15328 on aluminum, to provide better score protection on magnesium but to be slightly inferior in undercutting at the score on steel. One package wash primer C was equal to primer B on steel and aluminum but offered less protection on magnesium. The freshly prepared one package primer D was comparable to MIL-C-15328 on steel, and aluminum but had somewhat more corrosion along the score on magnesium. After 2 months storage the protective properties of primer D decreased.

Water immersion (Table VI) indicated the two component primer B to be inferior on aluminum to MIL-C-15328 and relatively comparable on steel. On magnesium both primers blistered. The one component wash primers were definitely inferior to the two component primers on all three substrates.

From the data, the one component polyamide-diphenolic acid wash primer does not perform satisfactorily, and further investigation appears unnecessary. The two component primer B, although superior to the one component primer and within the performance level of MIL-C-15328, offers no definite improvement over the existing specification wash primer.

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APPENDIX A

TABLE I

RATING SYSTEM FOR SALT SPRAY EXPOSURE

I. SCORE CONDITION

RATING	RUSTING AND/OR BLISTERING
5	None - 1/32 Inch
4	1/32 - 1/16 Inch
3	1/16 - 1/8 Inch
2	1/8 - 3/16 Inch
1	3/16 - 1/4 Inch
0	> - 1/4 Inch

II. UNDERCUTTING AT SCORE

RATING	
5	None - Intermittent
4	Continuous to 1/16 Inch
3	Continuous 1/16 - 1/8 Inch
2	Continuous 1/8 - 3/16 Inch
1	Continuous 3/16 - 1/4 Inch
0	Continuous > - 1/4 Inch

TABLE I - CONTINUED

III. SURFACE CONDITION*

RATING	A. RUSTING ALONE (CORROSION)
5	None
4	A.S.T.M. Photo No. 10 Type 1
3	A.S.T.M. Photo No. 9 Type 1
2	A.S.T.M. Photo No. 8 Type 1
1	A.S.T.M. Photo No. 7 Type 1
0	A.S.T.M. Photo No. 6 Type 1 Or Worse

RATING	B. RUSTING ACCOMPANIED BY BLISTERING
5	None
4	Trace, Less than 5 defects on 4x12 inch panel
3	A.S.T.M. Photo No. 8 Type 2
2	A.S.T.M. Photo No. 7 Type 2
1	A.S.T.M. Photo No. 6 Type 2
0	A.S.T.M. Photo No. 4 Type 2 Or Worse

RATING	C. BLISTERING ALONE
5	None
4	Trace-A.S.T.M. Blister Size 2 on 4x12 inch panel-2 Max. A.S.T.M. Blister Size 4 on 4x12 inch panel-4 Max. A.S.T.M. Blister Size 6 on 4x12 inch panel-6 Max. A.S.T.M. Blister Size 8 on 4x12 inch panel-8 Max.
3	A.S.T.M. Few - Record Blister Size
2	A.S.T.M. Medium - Record Blister Size
1	A.S.T.M. Medium-Dense - Record Blister Size
0	A.S.T.M. Dense - Record Blister Size

*Select Applicable Condition

TABLE I - CONTINUED

IV. SUBSTRATE CONDITION

RATING	PITTING AND/OR CORROSION SPOTS
5	None
4	Trace-A.S.T.M. Blister Size 2 on 4x12 inch panel-2 Max. A.S.T.M. Blister Size 4 on 4x12 inch panel-4 Max. A.S.T.M. Blister Size 6 on 4x12 inch panel-6 Max. A.S.T.M. Blister Size 8 on 4x12 inch panel-8 Max.
3	A.S.T.M. Few - Record Size
2	A.S.T.M. Medium - Record Size
1	A.S.T.M. Medium-Dense - Record Size
0	A.S.T.M. Dense - Record Size

AERATED WATER IMMERSION RATING SYSTEM

SURFACE CONDITION	BLISTERING
5	None
4	Trace - Record Blister Size-A.S.T.M. No.
3	Few - Record Blister Size-A.S.T.M. No.
2	Medium - Record Blister Size-A.S.T.M. No.
1	Medium-Dense - Record Blister Size-A.S.T.M. No.
0	Dense - Record Blister Size-A.S.T.M. No.

TABLE II

FORMULA A - CONTROL

MIL-C-15328 PRIMER, PRETREATMENT FOR METALS

COMPONENT I PIGMENTED POLYVINYL BUTYRAL

INGREDIENT	POUNDS	GALLONS
Polyvinyl Butyral	54.2	5.9
N-Butanol	121.0	17.9
Denatured Alcohol 190°	367.7	54.2
Basic Zinc Chromate	52.1	1.7
Lo-Micron Magnesium Silicate	7.7	0.3
Lampblack	0.6	---
	603.3	80.0

COMPONENT II ACID ACCELERATOR

85% Orthophosphoric Acid	30.8	2.0
Distilled Water	24.7	3.0
Denatured Alcohol 190°	101.6	15.0
	157.1	20.0

Mix 4 parts Component I with 1 part Component II by volume.

TABLE III

FORMULA B

TWO PACKAGE POLYAMIDE - DIPHENOLIC ACID RESIN PRIMER

COMPONENT I PIGMENTED RESIN

INGREDIENT	POUNDS	GALLONS
Polyamide-Diphenolic Acid Resin	88.0	10.6
99% Isopropanol	348.0	53.0
N-Butanol	20.0	3.0
Basic Zinc Chromate	89.0	2.8
Lo-Micron Magnesium Silicate	13.0	0.6
Lampblack	1.0	---
	559.0	70.0

COMPONENT II ACID ACCELERATOR

85% Orthophosphoric Acid	70.0	5.0
99% Isopropanol	99.0	15.0
N-Butanol	68.0	10.0
Toluol	144.0	20.0
	381.0	50.0

Mix 7 parts Component I with 5 parts Component II by volume.

TABLE IV

FORMULA C

ONE PACKAGE POLYAMIDE - DIPHENOLIC ACID RESIN PRIMER

RATIO OF 4 PARTS VEHICLE SOLIDS TO 1 PART ACID BY WEIGHT

INGREDIENT	POUNDS	GALLONS
Polyamide-Diphenolic Acid Resin	120.0	14.5
99% Isopropanol	480.0	73.0
N-Butanol	27.0	4.0
Basic Zinc Chromate	62.0	1.9
Strontium Chromate	62.0	1.8
Lo-Micron Magnesium Silicate	18.0	0.7

Charge into pebble mill and grind 4 hours. Premix the following and stir into mill. Continue grinding for 18 hours.

Distilled Water	23.4	2.7
85% Orthophosphoric Acid	35.0	2.5
	827.4	101.1

TABLE V

FORMULA D

ONE PACKAGE POLYAMIDE - DIPHENOLIC ACID RESIN PRIMER

RATIO 6 PARTS VEHICLE SOLIDS TO 1 PART ACID BY WEIGHT

INGREDIENT	POUNDS	GALLONS
Polyamide-Diphenolic Acid Resin	120.0	14.5
99% Isopropanol	480.0	73.0
N-Butanol	27.0	4.0
Basic Zinc Chromate	62.0	1.9
Strontium Chromate	62.0	2.0
Lo-Micron Magnesium Silicate	18.0	0.7

Charge into pebble mill and grind for 4 hours. Premix the following and stir into mill. Continue grinding for 18 hours.

Distilled Water	33.4	4.0
85% Orthophosphoric Acid	23.8	1.7
	826.2	101.8

TABLE VI

PHYSICAL PROPERTIES OF THE WASH PRIMERS

A. TWO COMPONENT PRIMERS	FORMULA A	FORMULA B
Grind - Hegman Gauge	6	6 1/2
Viscosity - Krebs Units Component I	60	51
% Non-Volatile - Admixed Primer	18.9	27.8
Pigment Volume Concentration - %	20.6	25.8
Package Condition After 2 Months Storage	Hard, Dry Sediment	Hard, Dry Sediment
B. ONE PACKAGE PRIMERS	**FORMULA C**	**FORMULA D**
Grind - Hegman Gauge	6 1/2	6
Initial Viscosity - Krebs Units	55	51
% Non Volatile	35.3	34.2
Pigment Volume Concentration - %	21.5	22.8
Viscosity After Storage - Krebs Units	Can and Glass (same)	Can and Glass (same)
24 Hours	57	51
1 Week	65	51
2 Weeks	87 - Seeding	53 - No Seeding
1 Month	95 - Seeding	53 - No Seeding
2 Months	Gummy	53 - No Seeding
4 Months	---	53 - No Seeding

TABLE VII

A. SALT SPRAY EXPOSURE TEST RESULTS

SUBSTRATE	STEEL					ALUMINUM					MAGNESIUM				
Formula	A	B	C	D	D 2 Months Storage	A	B	C	D	D 2 Months Storage	A	B	C	D	D 2 Months Storage
Hours Exposed	500	500	500	500	500	500	500	500	500	500	336	500	500	336	336
Rating - Score	4	4	4	4	4	5	5	5	5	4	1	2	0	0	0
Undercutting	4	3	3	4	3	5	5	5	5	4	5	5	5	5	5
Surface	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Substrate	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
B. AERATED WATER IMMERSION															
Hours Exposed	120	120	24	24	24	120	24	24	24	24	24	72	24	24	24
Surface Blistering	4	5	0	0	0	5	0	0	0	0	0	2	0	0	0
Size of Blister	<8	-	8	8	8	-	8	8	8	8	8	8	8	8	8

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